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IS 11995 (1987): Isoproturon, WP [FAD 1: Pesticides and Pesticides Residue Analysis]



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IS : 11995 - 1987

Indian Standard
**SPECIFICATION FOR
ISOPROTURON WP**

(First Reprint APRIL 1996)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR ISOPROTURON WP

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**AMENDMENT NO. 1 JULY 1994
TO
IS 11995 : 1987 SPECIFICATION FOR
ISOPROTURON EC**

(*Page 5, Table 1*) :

- a) *Sl No. (ii), col 2* }
b) *Sl No. (iii), col 2* } — Delete the words 'after accelerated storage'.

(*Page 5, Table 1, col 4, Heading*) — Substitute 'Appendix of IS 12004 : 1987' for 'Appendix of This Standard'.

(*Page 6, clause 4.1*) — Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause 2.4.1 of the standard.'

(*Page 6, Appendix A*) — Delete.

AMENDMENT NO. 2 MARCH 2002
TO
IS 11995 : 1987 SPECIFICATION FOR ISOPROTURON WP
*[Amendment No. 1, Title] — Substitute '**WP**' for '**EC**'.*

(FAD 1)

Reprography Unit, BIS, New Delhi, India

**AMENDMENT NO. 3 MAY 2002
TO
IS 11995 : 1987 SPECIFICATION FOR ISOPROTURON
WP**

[Page 4, clause 2.2 including Note] — Delete.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR ISOPROTURON WP

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 30 April 1987, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Isoproturon WP is used in the control of weeds in agricultural crops.

0.3 Isoproturon WP is generally manufactured to contain 50 percent or 75 percent (m/m) of isoproturon.

0.4 In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act, 1968* and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the Act and Rules, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for isoproturon WP.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a homogenous powder, off-white in colour and shall wet readily on mixing with water providing a suspension suitable for use as a spray.

*Rules for rounding off numerical values (*revised*).

2.2 Identity Test — Extract about 1 g of the sample in about 50 ml methanol. Centrifuge for about 5 minutes. Decant the clear supernatant liquid into a beaker. Add 250 to 300 ml of water to precipitate the isoproturon. Filter, wash thoroughly with water and dry at 100°C. Melting point of dry residue shall be between 154 and 156°C, and shall not be depressed by the addition of pure isoproturon. Further, IR spectrum of the residue taken in potassium bromide should be superimposable on the IR spectrum of the reference sample of isoproturon between 2.5 and 15 μ .

NOTE — Identity test need not be carried out if isoproturon content is determined by HPLC method (see A-1).

2.3 Isoproturon, technical employed in the formulation of this material shall conform to IS : 12004-1987*.

2.4 The material shall also comply with the requirements given in Table 1.

2.4.1 Isoproturon Content — When determined by the method prescribed in Appendix A, the observed isoproturon content percent (*m/m*) of any of the sample shall not differ from the declared nominal value by more than the tolerance limit given below:

<i>Nominal Value, Percent</i>	<i>Tolerance Limit, Percent</i>	
Up to 9	+ 10 — 5	} of the nominal value
Above 9 and below 50	± 5	
50 and above	+ 5 — 3	

2.4.1.1 The actual value of isoproturon content in the material shall be calculated to the second decimal place and then rounded off to the first decimal place before applying the tolerances given in 2.4.1.

2.4.1.2 The average content of all the samples taken shall not be less than the declared nominal content.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to the requirement given in IS : 8190 (Part 1)-1980†.

3.2 Marking — The container shall bear legibly and indelibly the following information and any other information as is necessary under the *Insecticides Act, 1968* and Rules framed thereunder:

- a) Name of the material;
- b) Name of the manufacturer or trade-mark;

*Specification for isoproturon, technical.

†Requirements for packing of pesticides: Part 1 Solid pesticide. (*first revision*).

- c) Date of manufacture and date of expiry;
- d) Batch number;
- e) Isoproturon content, percent (*m/m*);
- f) Net mass of the contents; and
- g) Cautionary notice as worded in *Insecticides Act, 1968* and Rules framed thereunder.

TABLE 1 REQUIREMENTS FOR ISOPROTURON WP
(*Clause 2.4*)

Sl. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix of this Standard	CI No. of IS : 6940-1982*
(1)	(2)	(3)	(4)	(5)
i)	Isoproturon content, percent by mass	Nominal value as declared on the container (<i>see 2.4.1</i>)	A	—
ii)	Sieving requirement, material passing through 75-micron IS Sieve, after accelerated storage, percent by mass, <i>Min</i> (<i>see Note 1</i>)	98.0	—	11.1
iii)	Suspensibility, after accelerated storage (<i>see Note 2</i>), percent by mass, <i>Min</i> :			
	a) for 50 percent WP	70.0	—	11.2
	b) for 75 percent WP	85.0	—	11.2
iv)	Wettability, in seconds, <i>Max</i>	120	—	11.4
v)	Acidity (as H_2SO_4), percent by mass, <i>Max</i>	0.10	—	11.3
	<i>or</i>			
	Alkalinity (as NaOH), percent by mass, <i>Max</i>	0.20	—	11.3

NOTE 1 — IS : 460 (Part 1)-1985 'Specification for test sieves: Part 1 Wire cloth test sieves (*third revision*)', BS Test Sieve 200, ASTM Test Sieve 200, Tyler Test Sieve 200, have their aperture within the limits specified for the above IS Test sieve and may, therefore, be used as 75-micron IS sieve.

NOTE 2 — The material shall not be subjected to accelerated storage treatment if it has crossed half of its shelf-life as ascertained from its date of manufacture and expiry date given on the container.

*Methods of test for pesticides and their formulations (*first revision*).

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3.3 BIS Certification Marking

The product may also be marked with Standard Mark.

3.3.1 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in IS : 10627-1983*.

5. TESTS

5.1 For suspensibility test, start with 0.5 percent of concentration after accelerated storage at $54 \pm 1^\circ\text{C}$ for 24 h as described in 11.2 of IS : 6940-1982†. Transfer 25 ml portion of suspension and sediment in a reaction flask. Wash the cylinder thrice with 100 ml of methanol. Proceed to determine active ingredient as described in Appendix A.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977‡) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

A P P E N D I X A

[*Table 1, Item (i)*]

DETERMINATION OF ISOPROTURON CONTENT

A-0. GENERAL

A-0.1 For the determination of isoproturon content, two methods, namely, HPLC (*see* A-1) and basic hydrolysis method (*see* A-2) have been prescribed. HPLC method will be the referee method in case of dispute.

*Methods for sampling of pesticidal formulations.

†Methods of test for pesticides and their formulations (*first revision*).

‡Specification for water for general laboratory use (*second revision*).

A-1. HPLC METHOD

A-1.1 Principle — A HPLC unit with a UV detector is used for the determination. Using a solution containing known amounts of the standard isoproturon and the internal standard, the response factor, *RF*, for isoproturon in the internal standard is arrived at. A solution containing the internal standard and active ingredient extracted quantitatively from a known mass of the sample (under test) is injected subsequently into the HPLC unit. The percentage of isoproturon in the sample is then computed by the standard relationship.

A-1.2 Apparatus

A-1.2.1 High performance liquid chromatograph equipped with a printer-plotter-cum integrator and UV detector. The suggestive HPLC operating conditions are given below. However, these operating conditions are likely to change with change in the HPLC equipment employed and are allowed, provided standardization is done:

Column	Silica 10 μm 25 cm \times 4.6 mm (S.S)
Solvent system	1) Cyclohexane 90 percent (v/v) 2) Isopropanol 10 percent (v/v)
Detector	UV (at 254 nm)
Solvent flow rate	1.5 ml/min
Chart speed	0.2 cm/min
Sample size	10 μl

A-1.2.2 Filtration Assembly — with G-4 sintered crucible.

A-1.3 Reagents

A-1.3.1 Internal Standard — Acetanilide, AR or equivalent grade.

A-1.3.2 Cyclohexane — Spectroscopic grade.

A-1.3.3 Isopropanol — Spectroscopic grade.

A-1.3.4 Isoproturon — of known purity.

A-1.4 Preparation of Standards and Calibration

A-1.4.1 Weigh accurately 0.25 g of acetanilide into a 100-ml volumetric flask and make up to volume using the cyclohexane-isopropanol mixture (90 : 10 v/v). This will give a solution containing 2.5 mg/ml of the internal standard.

A-1.4.2 Weigh 0.5 g of standard isoproturon into a 100-ml volumetric flask and dissolve it in 10 ml isopropanol. Make up to volume using

the cyclohexane. This will give a stock solution containing 5 mg/ml of the standard isoproturon. Pipette out 5 ml of this standard solution into a 50-ml volumetric flask. Then pipette out 5 ml of the internal standard solution into the same flask. Mix well. Make up to the mark using the solvent mixture. Call this *Solution A*.

A-1.5 Procedure

A-1.5.1 Weigh into a beaker with a glass rod, an amount of the test sample which will contain about 0.5 g of the active ingredient. Add about 10 ml of isopropanol and stir well for a few minutes. Warm the beaker to facilitate dissolution of the active ingredient. Filter carefully through a G-4 crucible and collect the filtrate in a conical flask. Wash the residue repeatedly with small quantities of cyclohexane, stir well and add these fractions to the G-4 crucible. Finally transfer the residue to the crucible and continue to wash with cyclohexane. Collect all the filtrate in the conical flask. Then quantitatively transfer it to a 100-ml volumetric flask and make up to volume with cyclohexane. Pipette out 5 ml of this final solution (containing the active ingredient extracted from the test sample) into a 50-ml volumetric flask. Pipette out 5 ml of internal standard solution (see A-1.4.1) into the same flask. Mix well and make up to the mark using the solvent mixture. Call this *Solution B*.

A-1.5.2 Introduce 10 μ l of the *Solution A* and *Solution B* into the HPLC unit. From the integrator, print out and note down the peak areas of the isoproturon and acetanilide peaks in both the cases. Adjust the attenuation in such a way that the isoproturon and acetanilide peaks are obtained within the scale in both the cases. (This attenuation may change from equipment to equipment). Compute the percentage of isoproturon content in the sample as given in A-1.6.

A-1.6 Calculations

$$\text{Isoproturon content, percent by mass} = \frac{m_1 \times A_2 \times P \times A_3}{A_1 \times m_2 \times A_4}$$

where

m_1 = mass of standard isoproturon in solution A;

A_2 = area of the isoproturon peak in solution B;

P = percentage purity of standard isoproturon;

A_3 = area of internal standard peak in solution A;

A_1 = area of the standard isoproturon peak in solution A;

m_2 = mass of the isoproturon sample taken for analysis; and

A_4 = area of internal standard peak in solution B.

A-1.7 Precision — Data obtained by this method indicate standard deviation of 0.7 and 0.6 for isoproturon at 50 percent and 75 percent level in the test samples respectively.

A-2. BASIC HYDROLYSIS METHOD

A-2.1 Principle

A-2.1.1 Isoproturon in the sample is hydrolyzed using potassium hydroxide in aqueous diethylene glycol (1:1 by volume). Dimethylamine, the volatile product of hydrolysis, is distilled off and absorbed in standard hydrochloric acid. From the quantity of hydrochloric acid consumed by dimethylamine, the percentage of isoproturon present in the original sample is computed.

A-2.2 Apparatus

A-2.2.1 An all glass distillation assembly of suitable type. A suggestive assembly is shown in Fig. 1. Minor changes in the suggestive assembly may be made to improve its suitability.

A-2.3 Reagents

A-2.3.1 *Hydrochloric Acid Solution* — 0.2 N.

A-2.3.2 *Sodium Hydroxide Solution* — 0.2 N.

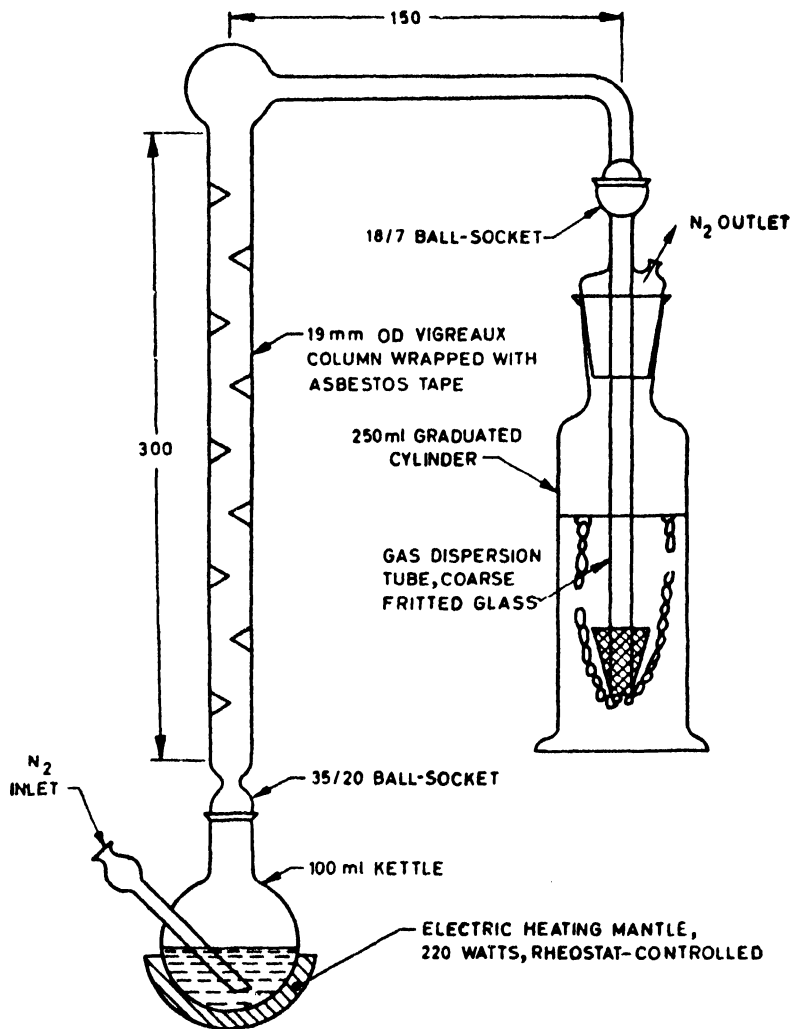
A-2.3.3 *Diethylene Glycol*

A-2.3.4 *Potassium Hydroxide Solution* — 35 percent aqueous.

A-2.3.5 *Silicon Defoamer*

A-2.4 Procedure

A-2.4.1 Accurately weigh an amount of the sample which will contain about 1.0 g of the active ingredient and transfer it quantitatively to a clean dry 500-ml reaction flask of the hydrolysis-cum-distillation apparatus. Add 50 ml of diethylene glycol and one or two boiling chips. One ml of silicon defoamer may be added to check foaming. Attach the reaction flask to the other parts of the assembly as shown in Fig. 1. 50 ml of hydrochloric acid is taken in a beaker in which the delivery tube and dips as shown in Fig. 1. Add 50 ml of 35 percent potassium hydroxide solution to the flask through a dropping funnel. Close the stopcock of the dropping funnel as soon as the addition of alkali is complete. Heat the contents of the reaction flask to boiling. Continue the distillation up to about 2½ hours. The distillation can be stopped when the contents of the flask become orange brown in colour. The distillate will cease to be alkaline by that time. This can further be confirmed using a multi-range pH paper to test the distillate. The pH of the distillate, as



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF ISOPROTURON CONTENT (ALKALINE HYDROLYSIS METHOD)

indicated by the colour of the pH paper, must be 7. Take care to disconnect the inverted funnel from the lower end of the condenser before switching off the heating source. If this is not done, the distillate will be sucked back into the distilling flask, when it starts cooling down, after the removal of the heating source. If however, arrangements can be made to maintain a flow of nitrogen gas through the distillation assembly, then the back suction problem will not be experienced. Rinse the funnel and the connecting tube with distilled water and add the rinsings to the receiver beaker. Titrate the contents of the beaker with 0.2 N sodium hydroxide solution using phenolphthalein as indicator. At the end point, the colourless solution will turn pink.

A-2.5 Calculations

$$\text{Isoproturon content, percent by mass} = \frac{[(V_1 \times N_1) - (V_2 \times N_2)] \times 20.6}{M}$$

where

V_1 = volume, in ml, of standard hydrochloric acid used;

N_1 = normality of the hydrochloric acid used;

V_2 = volume, in ml, of sodium hydroxide required to neutralize; the excess of acid when the sample is analyzed;

N_2 = normality of sodium hydroxide used for titration; and

M = mass, in g, of the sample taken for the test.

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